

**JP 2000-302490**

**English Abstract & Translation**

**From JPO Website**

**<http://www19.ipdl.inpit.go.jp/PA1/cgi-bin/PA1DETAIL>**

**&**

**<http://www4.ipdl.inpit.go.jp/Tokujitu/PAJdetail.ipdl?N0000=60&N0120=01&N2001=2&N3001=2000-302490>**

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : **2000-302490**

(43)Date of publication of application : **31.10.2000**

---

(51)Int.Cl.

C03C 27/12

B60J 1/00

---

(21)Application number : **11-112421**

(71)Applicant : **SEKISUI CHEM CO LTD**

(22)Date of filing : **20.04.1999**

(72)Inventor : **MIYAI JIRO**

**BANDO AKIHIKO**

---

## **(54) INTERMEDIATE FILM FOR LAMINATED GLASS AND LAMINATED GLASS**

### **(57)Abstract:**

**PROBLEM TO BE SOLVED:** To obtain both an intermediate for a laminated glass, having excellent adhesion ability and no reduction in penetration resistance and glass scattering resistance even in the case of use under high-moisture and high-humidity conditions and a laminated glass.

**SOLUTION:** This intermediate film for a laminated glass contains a polyvinyl acetal resin and a plasticizer and is set to have 1.5-7.5 kg/cm peel strength to show adhesivity between the intermediate film and a glass plate or 3-8 Pummel value to show adhesivity between the intermediate film and the glass plate. Especially an intermediate film for a safety glass comprising 100 pts.wt. of a polyvinyl butyral resin having 66-72 mol% butyralation degree and 30-50 pts.wt. of triethylene glycol di-2-ethylhexanoate. A laminated glass is obtained by using the intermediate film by a routine procedure.

---

**\* NOTICES \***

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. \*\*\*\* shows the word which can not be translated.

3. In the drawings, any words are not translated.

---

**CLAIMS**

---

[Claim(s)]

[Claim 1] The interlayer for glass laminates characterized by being an interlayer for glass laminates containing polyvinyl-acetal resin and a plasticizer, and the peel strength which expresses the adhesive property of an interlayer and a glass plate in 1 - 2 % of the weight of water content being 1.5-7.5kg/cm.

[Claim 2] The interlayer for glass laminates characterized by being the interlayer for glass laminates which contains TORIECHIRENGURIKORUJI-2-ethylhexanoate 20 - 60 weight sections whenever [ butyral-ized ] as % of the 66-72-mol polyvinyl-butyral-resin 100 weight section, and a plasticizer, and the peel strength which expresses the adhesive property of an interlayer and a glass plate in 1 - 2 % of the weight of water content being 1.5 - 7.5 kg/cm.

[Claim 3] The interlayer for glass laminates characterized by being an interlayer for glass laminates containing polyvinyl-acetal resin and a plasticizer, and the pan mel values which express the adhesive property of an interlayer and a glass plate in 1 - 2 % of the weight of water content being 3-8.

[Claim 4] The interlayer for glass laminates characterized by being the interlayer for glass laminates which contains TORIECHIRENGURIKORUJI-2-ethylhexanoate 20 - 60 weight sections whenever [ butyral-ized ] as % of the 66-72-mol polyvinyl-butyral-resin 100 weight section, and a plasticizer, and the pan mel values which express the adhesive property of an interlayer and a glass plate in 1 - 2 % of the weight of water content being 3-8.

[Claim 5] The glass laminate characterized by the interlayer for glass laminates of a publication being fastened by any 1 term of claims 1-4 between the glass plates of at least two sheets.

---

[Translation done.]

**\* NOTICES \***

JPO and INPIT are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. \*\*\*\* shows the word which can not be translated.

3. In the drawings, any words are not translated.

---

**DETAILED DESCRIPTION**

---

[Detailed Description of the Invention]

[0001]

[Field of the Invention] Especially this invention relates to the interlayer for glass laminates and a glass laminate excellent in adhesive ability.

[0002]

[Description of the Prior Art] The glass laminate with which the interlayer which consists of plasticization polyvinyl-acetal resin like plasticization polyvinyl butyral resin was fastened between the glass plates of at least two sheets is excellent in transparency, weatherability, penetration-proof, and the scattering tightness of glass, and is widely used for front windowpanes, such as an automobile, an aircraft, and a building.

[0003] Advanced penetration-proof and the scattering tightness of glass are demanded of this kind of glass laminate. In order to improve engine performance, such as this, it is necessary to adjust the adhesive strength of a glass plate and an interlayer moderately.

[0004] That is, at the glass laminate whose adhesive strength of a glass plate and an interlayer is too small, by the impact from the outside, it separates and disperses from an interlayer, and a glass plate and an interlayer are beaten by the impact from the outside by both the glass laminates whose adhesive strength of a glass plate and an interlayer is too large conversely, and glass penetrates with them.

[0005] The glass laminate with which the penetration-proof which comes to fasten the plasticization polyvinyl-butyr-al-resin interlayer which the alkali-metal salt or alkaline earth metal salt, and conversion silicone oil of monocarboxylic acid or dicarboxylic acid contained between the glass plates of at least two sheets has been improved is proposed by JP,53-18207,B.

[0006]

[Problem(s) to be Solved by the Invention] However, the glass laminate of the above-mentioned proposal does not have the enough adhesive property of an interlayer and a glass plate. That is, when such a glass laminate is used over a long period of time under an environment especially with much moisture and moisture, there is a problem that the adhesive ability of an interlayer and glass falls and penetration-proof and the scattering tightness of glass fall.

[0007] Even when it is used over a long period of time under an environment especially with much moisture and moisture, the place which this invention solves the above-mentioned problem and is made into the purpose is excellent in adhesive ability, and is to offer the interlayer for glass laminates and glass laminate with which penetration-proof and the scattering tightness of glass do not fall.

[0008]

[Means for Solving the Problem] In order to attain the above-mentioned purpose, in invention according to claim 1, the interlayer for glass laminates characterized by being an interlayer for glass laminates containing polyvinyl-acetal resin and a plasticizer, and the peel strength which expresses the adhesive property of an interlayer and a glass plate in 1 - 2 % of the weight of water content being 1.5 - 7.5 kg/cm is offered.

[0009] In invention according to claim 2, the interlayer for glass laminates characterized by being the interlayer for glass laminates which contains TORIECHIRENGURIKORUJI-2-ethylhexanoate 30 - 50 weight sections whenever [ butyral-ized ] as % of the 66-72-mol polyvinyl-butyral-resin 100 weight section and a plasticizer, and the peel strength which expresses the adhesive property of an interlayer and a glass plate in 1 - 2 % of the weight of water content being 1.5 - 7.5 kg/cm is offered.

[0010] In invention according to claim 3, it is an interlayer for glass laminates containing polyvinyl-acetal resin and a plasticizer, and the interlayer for glass laminates characterized by the pan mel values showing the adhesive property of an interlayer and a glass plate being 3-8 in 1 - 2 % of the weight of water content is offered.

[0011] In invention according to claim 4, it is the interlayer for glass laminates which contains TORIECHIRENGURIKORUJI-2-ethylhexanoate 30 - 50 weight sections whenever [ butyral-ized ] as % of the 66-72-mol polyvinyl-butyral-resin 100 weight section, and a plasticizer, and the interlayer for glass laminates characterized by the pan mel values showing the adhesive property of an interlayer and a glass plate being 3-8 in 1 - 2 % of the weight of water content is offered.

[0012] In invention of claim 5, the glass laminate characterized by the interlayer for glass laminates of a publication being fastened by any 1 term of claims 1-4 between the glass plates of at least two sheets is offered.

[0013] The polyvinyl-acetal resin which generally acetalizes polyvinyl alcohol by the aldehyde of carbon numbers 3-10, and is obtained as polyvinyl-acetal resin used for this invention is mentioned. Especially, 66-72-mol % of polyvinyl butyral resin is used suitably whenever [ butyral-ized ].

[0014] In order to obtain such polyvinyl-acetal resin, a well-known synthetic approach is adopted. For example, polyvinyl alcohol is dissolved in hot water, the obtained water solution is held at 0-95 degrees C, the aldehyde and acid catalyst of carbon numbers 3-10 are added to this, an acetalization reaction is advanced, subsequently, the temperature of the system of reaction is raised, it ripens, a reaction is completed, and the method of obtaining powder-like polyvinyl-acetal resin is adopted through neutralization, rinsing, and desiccation after that.

[0015] Here, as polyvinyl alcohol, the thing of average degree of polymerization 500-5000 is desirable, and the thing of average degree of polymerization 1000-2500 is still more desirable. When this average degree of polymerization is less than 500, the penetration-proof of a glass laminate may fall. On the contrary, when average degree of polymerization exceeds 5000, it is hard coming to carry out shaping of the resin film, and, moreover, the reinforcement of the resin film may become strong too much.

[0016] Moreover, as for the amount of residual acetyl groups of the resin obtained, it is desirable to consider as less than [ 30 mol % ] from the point of the transparency of an interlayer, thermal resistance, and weatherability, therefore, as for the saponification degree of polyvinyl alcohol, the thing beyond 70 mol % is used suitably. When the

saponification degree of polyvinyl alcohol is less than [ 70 mol % ], the transparency of resin and thermal resistance which are obtained may fall, or reactivity may fall. Here, the average degree of polymerization and saponification degree of polyvinyl alcohol are JIS. K It can measure based on 6726.

[0017] As an aldehyde of carbon numbers 3-10, the aldehyde of aliphatic series, such as propionaldehyde, n-butyraldehyde, isobutyraldehyde, a valeraldehyde, n-hexyl aldehyde, 2-ethyl butyraldehyde, n-heptyl aldehyde, n-octyl aldehyde, n nonyl aldehyde, n-decyl aldehyde, a benzaldehyde, and a cinnamaldehyde, or aromatic series is mentioned.

Aldehydes, such as this, are used combining independent or two sorts or more.

[0018] When the carbon number of an aldehyde is less than three, the moldability of the resin film is sometimes inadequate. On the contrary, when the carbon number of an aldehyde exceeds 10, the reactivity of acetalization may fall, or moreover, it becomes easy to generate the block of resin during a reaction, and difficulty may be followed on composition of resin. The aldehyde of 4-8 is more preferably used for a carbon number. It is desirable to use the polyvinyl butyral resin especially acetalized and obtained by n-butyraldehyde. Polyvinyl butyral resin is excellent in transparency, an adhesive property, or weatherability, and, moreover, manufacture of resin is easy for it.

[0019] Generally the degree of acetalization of polyvinyl-acetal resin is made into 40-75-mol %. Compatibility with a plasticizer falls and the degree of acetalization of resin stops easily being able to mix a plasticizer required for reservation of penetration-proof less than [ 40 mol % ]. On the contrary, if the degree of acetalization exceeds 75-mol %, the reinforcement of an interlayer may fall, or reaction time of long duration may be required, and it is not desirable from a composite point.

[0020] In the case of polyvinyl butyral resin, 66-72-mol% of especially the degree of acetalization (whenever [ butyral-ized ]) is desirable, and it is more preferably made into 67.5-70-mol %. When an interlayer becomes [ whenever / butyral-ized ] easy to absorb moisture less than [ 66 mol % ], milkiess of the periphery section of a glass laminate becomes easy to take place and whenever [ butyral-ized ] exceeds 72-mol % conversely, the reinforcement of an interlayer may fall. Here, the degree of acetalization (whenever [ butyral-ized ]) and the amount of residual acetyl groups of polyvinyl-acetal resin are JISK. It can measure based on 6728 or a nuclear magnetic resonance method (NMR).

[0021] As a plasticizer, phosphoric-acid system plasticizers, such as organic system plasticizers, such as the well-known plasticizer used for this kind of interlayer, for example, monobasic-acid ester, and polybasic acid ester, and an organic phosphorus acid system, an organic phosphorous acid system, etc. are used. Especially, TORIECHIRENGURIKORUJI-2-ethylhexanoate 20 - 60 weight sections are used to the 66-72-mol % polyvinyl-butyral-resin 100 weight section whenever [ butyral-ized ].

[0022] In monobasic-acid ester, the glycol system ester obtained by the reaction with organic acids, such as triethylene glycol, butanoic acid and an isobutyric acid, a caproic acid, 2-ethyl butanoic acid, oenanthic acid, n-octylic acid, a 2-ethylhexyl acid, pelargonic acid (n-nonylic acid), and a DESHIRU acid, for example is desirable. In addition, the ester of tetraethylene glycol, tripropylene glycol, and the organic acid like the above is also used.

[0023] As polybasic acid ester, the shape of a straight chain of organic acids, such as an adipic acid, sebacic acid, and an azelaic acid, and carbon numbers 4-8 and ester with branching-like alcohol are desirable, for example. Moreover, as a phosphoric-acid system

plasticizer, TORIBUTOKISHI ethyl phosphate, isodecyl phenyl phosphate, triisopropyl phosphite, etc. are desirable.

[0024] Especially, triethylene glycol di-2-ethyl butyrate, TORIECHIRENGURIKORUJI-2-ethylhexanoate, triethylene glycol dicaprylate, triethylene glycol-G n-octanoate, triethylene glycol-G n-heptanoate, tetraethylene glycol-G n-heptanoate, other dibutyl sebacate, a 2-ethylhexyl horse mackerel peat, and a dibutyl carbitol horse mackerel peat are used suitably.

[0025] Also in it, TORIECHIRENGURIKORUJI-2-ethylhexanoate is the optimal. This TORIECHIRENGURIKORUJI-2-ethylhexanoate is obtained by carrying out the esterification reaction of triethylene glycol and the 2-ethylhexyl acid more than that amount of 2 double using a catalyst. The engine performance stabilized since there was little volatility is obtained, and, moreover, this TORIECHIRENGURIKORUJI-2-ethylhexanoate is excellent also in respect of the work environment in the case of melting kneading, and excellent also in the trim cut nature in the case of doubling processing of an interlayer, and excellent also in the low-temperature flexibility of an interlayer further.

[0026] Plasticizers, such as this, are 40 - 60 weight sections preferably [ 20 - 70 weight \*\*\*\*\* / \*\* ] to the polyvinyl-acetal resin 100 weight section, and more preferably. There is \*\* to which the penetration-proof of a glass laminate will fall if a plasticizer is less than 20 weight sections, conversely, when a plasticizer exceeds 70 weight sections, a plasticizer may ooze out from an interlayer and the adhesive property and transparency of a glass laminate may fall.

[0027] Moreover, the well-known additives including an adhesive strength regulator currently used for interlayers for glass laminates, such as an ultraviolet ray absorbent, light stabilizer, an antioxidant, and a coloring agent, can be blended if needed.

[0028] As an adhesive strength regulator, potassium salt and magnesium salt of an organic carboxylic acid, such as formic acid, an acetic acid, a propionic acid, 2-ethyl butanoic acid, an octanoic acid, a 2-ethylhexyl acid, oxalic acid, butanoic acid, and a benzoic acid, are used, for example. In addition, the metal salt of organic acids, such as a hydrochloric acid and a nitric acid, is also used.

[0029] The magnesium salt of the especially above organic carboxylic acids is suitable. Such magnesium salt exists in the form of a salt, without ionizing in an interlayer, by drawing a water molecule near, is considered to suppress the fall of the adhesive strength between a glass plate and an interlayer, and can make penetration-proof of the glass laminate obtained good. Moreover, since magnesium salt, such as this, is distributed over a film front face at high concentration, without condensing in an interlayer, it is little and shows the adhesive strength adjustment effectiveness. Moreover, too much milkiness of the periphery section of the glass laminate by moisture absorption is not caused.

[0030] As an ultraviolet ray absorbent, the thing P of a benzotriazol system, for example, Ciba-Geigy tinuvin, (trade name), tinuvin 320 (trade name), tinuvin 326 (trade name), tinuvin 328 (trade name), etc. are used, for example. As light stabilizer, the thing by the Asahi electrification company of a hindered amine system, ADEKA stub LA-57 [ for example, ] etc., (trade name) is used. As an antioxidant, the thing BHT by Sumitomo Chemical Co., Ltd. of a phenol system, for example, Sumi Reiser, (trade name), Ciba-Geigy IRUGA NOx 1010 (trade name), etc. are used.

[0031] The plasticizer of requirements can be blended with the above-mentioned polyvinyl-acetal resin, other additives can be blended further if needed, the interlayer of

this invention carries out kneading fusion of this with an extruder, and it can be fabricated in the shape of a sheet and it can obtain it. Moreover, after carrying out kneading fusion, it can also obtain by carrying out a roll press. The thickness of an interlayer is decided in consideration of penetration-proof required as a glass laminate etc. It is desirable to be especially referred to as 0.2-2mm.

[0032] By carrying out a deer and selecting suitably the class and amounts of an additive, such as an adhesive strength regulator blended the class of polyvinyl-acetal resin, the class of an amount and plasticizer, an amount, and if needed, in invention claim 1 and given in two, in 1 - 2 % of the weight of water content, it is set up so that the peel strength showing the adhesive property of an interlayer and glass may serve as 1.5 - 7.5 kg/cm.

[0033] Here, the above-mentioned peel strength is measured as follows. First, a glass laminate is manufactured using the interlayer which adjusted water content to 1 - 2% of the weight. In this way, generally the water content of the interlayer in the glass laminate obtained becomes 1 - 2 % of the weight comparable as the interlayer in front of doubling processing. In order to measure the water content of the interlayer in a glass laminate, the dielectrometry using the dielectric constant of the near infrared ray absorption analysis using a water absorption band or water is adopted suitably. In this case, it is convenient to create the calibration curve showing the relation between the water content of the interlayer to be used and the water content of the interlayer in the glass laminate obtained.

[0034] Next, cut into width of face of 1cm, and die length of 10cm the glass laminate obtained above, and it considers as a test piece. Remove the glass plate of that one side, make the end of an interlayer exfoliate beforehand, and this edge that exfoliated is made to pull and exfoliate at 90 degrees using a tension tester the condition for 22-degree-C 500mm [ in  $\pm 3$  degrees C and exfoliation rate ], it tears off at that time, and the force is measured, and let this value be peel strength (kg/cm).

[0035] In this case, since it will become easy to remove a glass plate and to make the end of an interlayer exfoliate beforehand if the glass laminate is manufactured on both sides of polyester film (PET film) in the predetermined location between an interlayer and a glass plate in case a glass laminate is manufactured, it is convenient for creation of a test piece to adopt this approach. In addition, in case an interlayer is torn off from a glass plate, when an interlayer fractures, it is in the condition which reinforced the interlayer with the above-mentioned polyester film (PET film), and tears off from a glass plate.

[0036] Moreover, in invention claim 3 and given in four, by selecting suitably the class and amounts of an additive, such as an adhesive strength regulator blended the class of polyvinyl-acetal resin, the class of an amount and plasticizer, an amount, and if needed, in 1 - 2 % of the weight of water content, it is set up so that the pan mel value showing the adhesive property of an interlayer and glass may be set to 3-8.

[0037] Here, the above-mentioned panmel value is measured as follows. First, the glass laminate whose water content of the interlayer in a glass laminate is 1 - 2 % of the weight as mentioned above is manufactured. Next, 16 timing of this glass laminate is carried out to -18-degree-C temperature of  $\pm 0.6$  degrees C, and the hammer whose head is 0.45kg strikes the center section (150mm long and 150mm wide part) of this glass laminate, it grinds until the particle size of glass is set to 6mm or less, and whenever [ exposure-Ushiro's film in which glass carried out partial avulsion ] is measured, and it expresses with the pan mel value (Sekisui law) by the following table 1.



[0038]

[Table 1]

中間膜の露出度 (%)	パンメル値
1 0 0	0
9 0	1
8 5	2
6 0	3
4 0	4
2 0	5
1 0	6
5	7
2 以下	8

[0039] Measuring each of claim 1, peel strength in invention given in two, claims 3, and pan mel values in invention given in four in the range whose water content of an interlayer is 1 - 2 % of the weight is based on the following reason. That is, although the interlayer currently fastened to the glass laminate absorbed moisture while in use, the water content of an interlayer found out that adhesive strength with glass became the lowest in the range which is 1 - 2 % of the weight. Moreover, although the interlayer currently fastened to the glass laminate was absorbed moisture and swollen while in use, that the swelling rate becomes easy to cause exfoliation most greatly found out that it was the range whose water content is 1 - 2 % of the weight.

[0040] And it sets to invention claim 1 and given in two. If it is set up so that the peel strength from which the water content of an interlayer serves as an adhesive index of an interlayer and a glass plate in the range which is 1 - 2 % of the weight may serve as 1.5 - 7.5 kg/cm Even if it used it over the long period of time under the environment especially with much moisture and moisture, it found out that the fall of the penetration-proof by the fall of the adhesive ability of an interlayer and glass and the scattering tightness of glass came to be hard for being generated. That is, since adhesive strength is too strong when it becomes easy to generate exfoliation between an interlayer and a glass plate under a high-humidity condition and peel strength exceeds 7.5 kg/cm conversely, since adhesive strength is too weak when peel strength is less than 1.5 kg/cm, an interlayer and a glass plate are broken into coincidence and it becomes easy for shock resistance and penetration-proof to fall.

[0041] Moreover, it sets to invention claim 3 and given in four. If it is set up so that the pan mel value from which the water content of an interlayer serves as an adhesive index of an interlayer and a glass plate in the range which is 1 - 2 % of the weight may be set to 3-8 Even if it used it over the long period of time under the environment especially with much moisture and moisture, it found out that it was hard coming to generate the fall of the penetration-proof by the fall of the adhesive ability of an interlayer and a glass plate, and the scattering tightness of glass. That is, since adhesive strength is too strong when it becomes easy to generate exfoliation between an interlayer and a glass plate under a

high-humidity condition and a pan mel value exceeds 8 conversely, since adhesive strength is too weak when a pan mel value is less than 3, an interlayer and glass are broken into coincidence and it becomes easy for shock resistance and penetration-proof to fall.

[0042] In this way, in order to manufacture a glass laminate according to claim 5 using the interlayer for glass laminates of this invention obtained, preparative pressure arrival and this sticking by pressure are performed like the process of the usual glass laminate, and it is manufactured.

[0043] Preparative pressure arrival sandwiches an interlayer between the transparent inorganic glass plates of two sheets. This layered product to a nip roll Through, For example, the approach of carrying out preparative pressure arrival, while drawing through and deaerating on conditions with a pressure [ of about 2-10kg/cm<sup>2</sup> ], and a temperature of about 40-100 degrees C (drawing through the rolling method), Or the above-mentioned layered product is put into the rubber back, a rubber bag is connected to an exhaust air system, and it is [ about ]. -Temperature is raised to the vacuum (absolute pressure 360 - 10mmHg) of 400 - -750mmHg, carrying out suction reduced pressure, and the approach (the vacuum bag method) of carrying out preparative pressure arrival at about 40-100 degrees C is adopted.

[0044] Subsequently, a press is used for the layered product by which preparative pressure arrival was carried out with a conventional method, using an autoclave, and it is the temperature of about 120-150 degrees C, and about 2-15kg/cm<sup>2</sup>. Actual sticking by pressure is carried out by the pressure. In this way, the glass laminate of this invention is obtained.

[0045] In addition, as the above-mentioned glass plate, not only an inorganic glass plate but organic glass plates, such as a polycarbonate plate and a polymethylmethacrylate plate, can be used. Moreover, the laminating configuration of a glass laminate can be considered as a multilayer configuration not only like for example, 3 lamination of a glass plate / interlayer / glass plate but a glass plate / interlayer / glass plate / interlayer / glass plate.

[0046]

[Embodiment of the Invention] Hereafter, the example and the example of a comparison of this invention are shown.

(Examples 1-8 and examples 1-5 of a comparison)

Whenever [ < butyral-ized ], supplying and stirring the 99.2-mol % polyvinyl alcohol 198 weight section (4.5 mol considerable amount of vinyl alcohol) the ion-exchange-water 2900 weight section, average degree of polymerization 1700, and whenever [ saponification ], it heated at 95 degrees C and dissolved in the reactor with synthetic > stirring equipment of the polyvinyl butyral resin of the shape of powder of the 0.8 mol % and the average degree of polymerization 1700 of 65-mol % and survival acetyl groups. After cooled this water solution at 30 degrees C, having added the n-butyraldehyde 143 weight section (2.0 mols) and the 35% hydrochloric-acid 208 weight section (2.1 mols) to this, lowering solution temperature subsequently to 2 degrees C, holding this temperature and polyvinyl butyral resin's depositing, the temperature up of the solution temperature was carried out to 30 degrees C, and it was held for 5 hours. Then, the sodium-carbonate 156 weight section (1.8 mols) was added, it neutralized, rinsing and desiccation were performed, and the polyvinyl butyral resin of the shape of powder of the 0.8 mol % and

the average degree of polymerization 1700 of 65-mol % and survival acetyl groups was obtained whenever [ butyral-ized ].

[0047] It sets whenever [ < butyral-ized ] to composition of synthetic > polyvinyl-butyral-resin A of the polyvinyl butyral resin of the shape of powder of the 0.8 mol % and the average degree of polymerization 1700 of 68-mol % and survival acetyl groups. It replaces with the n-butyraldehyde 143 weight section (2.0 mols), and the n-butyraldehyde 149 weight section (2.09 mols) is used. Except it It carried out like composition of polyvinyl-butyral-resin A, and the polyvinyl butyral resin of the shape of powder of the 0.8 mol % and the average degree of polymerization 1700 of 68-mol % and survival acetyl groups was obtained whenever [ butyral-ized ].

[0048] It sets whenever [ < butyral-ized ] to composition of synthetic > polyvinyl-butyral-resin A of the polyvinyl butyral resin of the shape of powder of the 0.8 mol % and the average degree of polymerization 1700 of 69-mol % and survival acetyl groups. It replaces with the n-butyraldehyde 143 weight section (2.0 mols), and the n-butyraldehyde 152 weight section (2.12 mols) is used. Except it It carried out like composition of polyvinyl-butyral-resin A, and the polyvinyl butyral resin of the shape of powder of the 0.8 mol % and the average degree of polymerization 1700 of 69-mol % and survival acetyl groups was obtained whenever [ butyral-ized ].

[0049] Magnesium acetate, 2-ethyl butanoic acid magnesium, octanoic-acid magnesium, potassium acetate, and a calcium chloride are used as triethylene glycol di-2-ethyl butyrate (3GH), TORIECHIRENGURIKORUJI-2-ethylhexanoate (3GO), di(2-ethylhexyl) adipate, and an adhesive strength regulator as the <production of interlayer> above-mentioned three kinds of polyvinyl butyral resin, and a plasticizer. Raw materials, such as this, were mixed as shown in Table 1, and 13 kinds (eight kinds of examples, five kinds of examples of a comparison) of mixture was obtained.

[0050] Subsequently, it is the kneading object obtained by supplying each mixture, such as this, to a roll mill, and carrying out melting kneading by the press-forming machine 150 degrees C and 100kg/cm<sup>2</sup> Press forming was carried out for 30 minutes on conditions, and 13 kinds (eight kinds of examples, five kinds of examples of a comparison) of interlayers were obtained by 0.76mm in thickness.

[0051] <Production of glass laminate> (production of glass laminate of examples 1-5 and examples 1 and 2 of comparison) above-mentioned each interlayer is put into an air conditioned room. After adjusting so that water content may become 1 - 2% of the weight, it judges to 300mm by 300mm. This is fastened between the float glasses (2.5mm in 300mm[ 300mm by ] x thickness) of two sheets. Preparative pressure arrival is carried out drawing through and deaerating this fastening object on conditions with through, a pressure [ of 5kg/cm<sup>2</sup> ], and a temperature of 80 degrees C to a nip roll, and it is this within an autoclave 135 degrees C and 12kg/cm<sup>2</sup> Actual sticking by pressure was carried out on conditions, and seven kinds (five kinds of examples, two kinds of examples of a comparison) of glass laminates were obtained.

[0052] Each above-mentioned interlayer is put into an air conditioned room. (Production of the glass laminate of examples 6-8 and the examples 3-5 of a comparison) After adjusting so that water content may become 1 - 2% of the weight, it judges to 300mm by 300mm. This is fastened between the float glasses (2.5mm in 300mm[ 300mm by ] x thickness) of two sheets. Put this fastening object into a vacuum bag, and it holds for 20 minutes by degree of vacuum 20torr. Within oven, it holds for 30 minutes, preparative

pressure arrival is carried out at 90 degrees C, making it a vacuum, this is taken out from a vacuum bag, and they are 150 degrees C and 13kg/cm<sup>2</sup> within an autoclave. Actual sticking by pressure was carried out on conditions, and six kinds (three kinds of examples, three kinds of examples of a comparison) of glass laminates were obtained.

[0053] <the performance evaluation of a glass laminate> -- when the water content of an interlayer was measured by the near infrared ray absorption analysis using a water absorption band about glass laminates, such as this, it was 1 - 2% of the weight of the range altogether. This water content set the glass laminate to the near-infrared spectrometer (Ubest V-570 by Jasco Corp.), measured the spectrum, and, specifically, measured it by the peak (water absorption band) of 1925cm<sup>-1</sup>, and the intensity ratio of 1705cm<sup>-1</sup> (absorption band of polyvinyl butyral resin).

[0054] Moreover, the friction test, the pan mel trial, the dustability trial of glass, and the penetration-proof trial were performed by the following approach about glass laminates, such as this. Moreover, the volatile trial of a plasticizer was performed by the following approach about the above-mentioned interlayer. Test results, such as this, are shown in Table 2 and 3.

[0055] (1) Cut a friction test glass laminate into width of face of 1cm, and die length of 10cm, and consider as a test piece. Remove the glass of that one side, make the end of an interlayer exfoliate beforehand, and this test piece is attached at a level with the tension tester [tensilon UCE500 (cage en tech company make)] lower grip. Attached in the up grip the end of the interlayer made to exfoliate beforehand, and pull at 90 degrees, they was made to exfoliate the condition for 22-degree-C 500mm [ in \*\*3 degrees C and exfoliation rate ], it tore off at that time, and the force (kg/cm) was measured.

[0056] In addition, when the test piece used for the friction test manufactured a glass laminate, specially, it manufactured the glass laminate on both sides of polyester film (PET film) in the predetermined location between an interlayer and a glass plate, and created the predetermined test piece from this glass laminate.

[0057] (2) Pan mel trial (Sekisui law)

16 timing of the glass laminate was carried out to -18-degree-C temperature of \*\*0.6 degrees C, and the hammer whose head is 0.45kg struck the center section (150mm long and 150mm wide part) of this glass laminate, it ground until the particle size of glass was set to 6mm or less, and the pan mel value (Sekisui law) showed whenever [ exposure- Ushiro's film in which glass carried out partial avulsion ] by Table 1.

[0058] (3) About the dustability trial glass laminate of glass, the above-mentioned pan mel trial (Sekisui law) was performed, the pan mel values 3-8 were considered as success (O), and except [ its ] was shown as rejection (x).

[0059] (4) Penetration-proof trial (based on JIS R 3212)

16 timing of the glass laminate was carried out to the temperature of 23 degree-C\*\*\*\*, where it fixed to the housing and the periphery section of this glass laminate is held horizontally, the center section of the glass laminate was made to carry out free fall of the 2.26kg shot from on that, and the drop height (m) in which a test piece can prevent penetration of a shot was measured. Drop height of 4m or more was considered as success (O), and less than [ it ] was shown as rejection (x).

[0060] (5) The volatile interlayer of a plasticizer was left in 150-degree C oven for 1 hour, and was heated in it, the weight of the interlayer before heating and after heating was measured, and the percentage showed Shigekazu (heating Saki [ Shigekazu ]-heating

Shigekazu Ushiro) / heating Saki.

[0061]

[Table 2]

		実 施 例					比 較 例	
		1	2	3	4	5	1	2
中間膜の組成（重量部）	ポリビニルアセタール樹脂							
	剥離剤							
	剥離剤	100	100	100	100	100	100	100
	アセチル化度(%)	65	65	65	68	68	65	68
	残存アセチル基(%)	0.8	0.8	0.8	0.8	0.8	0.8	0.8
	平均重合度	1700	1700	1700	1700	1700	1700	1700
	可塑剤							
	トリエチレングリコール-2	40	40	40	—	—	40	—
	エチレングリコール	—	—	—	38	38	—	38
	トリエチレングリコール-2	—	—	—	38	38	—	38
	エチレングリコール	—	—	—	—	—	—	—
	接着性調整剤							
	酢酸マグネシウム	0.04	—	—	—	0.04	—	—
	2-エチル 酢酸マグネシウム	—	0.04	—	0.04	0.04	—	—
性能評価	塩化カルシウム	—	—	0.05	—	—	—	—
	酢酸カルシウム	—	—	—	0.03	—	—	0.09
	・含水率1～2重量%における剥離強度 (kg/cm)	2.6～4.1	3.5～6.1	3.1～5.5	1.7～3.8	4.5～7.2	7.8～9.2	0.4～1.2
	・ガラスの飛散性	○	○	○	○	○	○	×
	・耐貫通性	○	○	○	○	○	×	○

[0062]

[Table 3]

		実施例			比較例		
		6	7	8	3	4	5
中間膜の組成(重量部)	ポリビニルアセタール樹脂						
	ポリビニルアルコール樹脂	100	100	100	100	100	100
	アルコール化度(%)	69	69	69	69	69	69
	残存アセチル基(%)	0.8	0.8	0.8	0.8	0.8	0.8
	平均重合度	1700	1700	1700	1700	1700	1700
	可塑剤						
	トリエチレングリコール-2	39	39	39	—	39	39
	エチルヘキサノート						
	アジピン酸-2エチルヘキシル	—	—	—	39	—	—
	接着性調整剤						
	酢酸ブチル	—	—	—	—	—	—
	2-エチル 酢酸ブチル	0.03	—	0.02	0.03	—	—
性能評価	ウレタン酸ブチル	—	0.04	0.05	—	—	—
	酢酸ブチル	—	—	—	—	0.09	0.09
	・含水率1~2重量%におけるパンメル値	4.5 ~ 7.5	3.5 ~ 7.5	4.5 ~ 7.5	4.5 ~ 7.5	8.0 ~ 9.5	1.0 ~ 2.0
	・ガラスの飛散性	○	○	○	○	○	×
	・耐貫通性	○	○	○	○	×	×
	落球高さ(m)	7.0	7.0	7.0	7.0	3.5	3.5
	・加熱減量(wt%)	1	1	2	8	1	1

[0063]

[Effect of the Invention] As above-mentioned, invention according to claim 1 is an interlayer for glass laminates containing polyvinyl-acetal resin and a plasticizer, and is set to 1 - 2 % of the weight of water content. The glass laminate which the peel strength showing the adhesive property of an interlayer and glass is 1.5 - 7.5 kg/cm, and is obtained using such an interlayer Even when it is used over a long period of time under an environment especially with much moisture and moisture, it excels in adhesive ability and the remarkable effectiveness that penetration-proof and the scattering tightness of glass do not fall is done so.

[0064] Moreover, invention according to claim 3 is an interlayer for glass laminates containing polyvinyl-acetal resin and a plasticizer, and is set to 1 - 2 % of the weight of water content. The glass laminate which the pan mel values showing the adhesive property of an interlayer and glass are 3-8, and is obtained using such an interlayer Even when it is used over a long period of time under an environment especially with much moisture and moisture, it excels in adhesive ability and the remarkable effectiveness that penetration-proof and the scattering tightness of glass do not fall is done so.

[0065] And it is the interlayer for glass laminates to which invention contains TORIECHIRENGURIKORUJI-2-ethylhexanoate 20 - 60 weight sections whenever [ butyral-ized ] in claim 2 and four publications as % of the 66-72-mol polyvinyl-butyral-resin 100 weight section, and a plasticizer. The engine performance stabilized since there was little volatility of a plasticizer is obtained, and it sets to manufacture of an interlayer.

The remarkable effectiveness of excelling also in respect of the work environment in the case of melting kneading of a combination constituent, and excelling also in the trim cut nature in the case of doubling processing of an interlayer, and excelling also in the low-temperature flexibility of an interlayer further, and excelling also in transparency, an adhesive property, or weatherability is done so.

---

[Translation done.]